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PATENT

Docket No. 150.0045072

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**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

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Applicant(s):	Hineman et al.	)	Group Art Unit:	1751
		)		
Serial No.:	09/935,234	)	Examiner:	Webb, G.
		)		
Filed:	August 22, 2001	)	Confirm. No.:	9323
For:	DILUTE CLEANING COMPOSITION AND METHOD FOR USING SAME			

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**APPEAL BRIEF**

Mail Stop Appeal Brief - Patents  
Commissioner for Patents  
P. O. Box 1450  
Alexandria, VA 22313-1450

Dear Sir:

Applicants present this Appeal Brief in support of the appeal from the final rejections of claims 64-70 of the above-identified patent application as indicated in the Notice of Appeal filed April 30, 2003.

**Real Party In Interest**

The real party in interest is Micron Technology, Inc. of Boise, Idaho, as evidenced by the assignment recorded at Reel 10017/Frame 0055 of the parent application (U.S. Serial No. 09/325,375 filed June 04, 1999). That assignment also effectively assigns the rights in this divisional patent application to Micron Technology, Inc.

**Related Appeals and Interferences**

There are no known related appeals or interferences pending in connection with the present application.

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## Status of Claims

Claims 64-70 are pending, with claims 1-63 having been canceled upon the filing of the patent application. The final rejection of claims 64-70 is appealed.

## Status of Amendments

The amendment to claim 67 and the accompanying remarks provided in the Amendment and Response filed December 16, 2002, were considered but not deemed to place claims 64-70 in condition for allowance (*see* Final Office Action dated January 31, 2003). All of pending claims 64-70 are presented in attached Appendix A.

## Summary of the Invention

The present invention provides a chemical composition for use in wet cleans in semiconductor processing which includes both phosphoric acid and acetic acid. The chemical composition preferably includes about 5% by volume or less of phosphoric acid with the remaining portion of the composition being acetic acid or with the remaining portion being acetic acid and deionized water. In other words, the composition includes about X% by volume or less of phosphoric acid and about (100-X)% by volume or less of acetic acid, where X is 5. This chemical composition provides better solvation of metallized polymers and organic polymers than previously used cleaning compositions, such as standard phosphoric acid cleans. However, the chemical composition prevents removal of too much material from the surface being cleaned, e.g., an aluminum containing surface. The use of both acidic components together in the cleaning composition is beneficial. The phosphoric acid component promotes solvation of inorganic residue, while the acetic acid component promotes solvation of organic residue. Further, and surprisingly, acetic acid passivates a metal containing surface being cleaned, e.g., patterned aluminum, so as to prevent undesirable removal of substantial amounts of the metal by the phosphoric acid during the cleaning process, (see page 6, lines 9-25).

Preferably, the cleaning composition is a dilute composition (a composition having about 35% reactive components or less by volume). Such a composition is advantageous as compared

with previously used strong (i.e., not dilute) solutions because it does not require special hazardous waste disposal. *See* page 6, lines 26-29.

### Issues

- I. Whether claims 64-70 are properly rejected under 35 U.S.C. § 102(b) as being anticipated by Oles (U.S. Patent No. 4,145,451).
- II. Whether claims 64-70 are properly rejected under 35 U.S.C. § 102(b) as being anticipated by Baker (U.S. Patent No. 5,508,229).
- III. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Hitachi (JP 77048059) in view of Hwang et al. (U.S. Patent No. 4,681,657). [It is noted that the Final Office Action states that "[c]laims 64-70 remain rejected under 35 USC 103 as being unpatentable over Hitachi." However, since a 35 U.S.C. § 103(a) rejection of these claims in view of Hitachi has never been made, it is assumed that the Examiner is referring to the previous rejection over Hitachi *in view of* Hwang et al.]
- IV. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Petzow (*Metallographic Etching*) in view of Hwang et al.
- V. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Koike et al. (U.S. Patent No. 4,256,520) in view of Hwang et al.
- VI. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Martin et al. (U.S. Patent No. 4,230,522) in view of Hwang et al.

The previously-issued obviousness-type double patenting rejection of claims 64-70 over claims 1-5 of U.S. Pat. No. 6,384,001 was not repeated in the final Office Action and is thus considered to be withdrawn. In the event this rejection was excluded by error, or in the event the rejection is otherwise reinstated, Applicants choose not to appeal this issue at this time. Any decision to file a terminal disclaimer, however, will be deferred until otherwise allowable subject matter is identified.

## Grouping of Claims

For the purposes of this appeal, claims 64-66 stand or fall together and claims 67-70 stand or fall together.

## Arguments

### **I. Whether claims 64-70 are properly rejected under 35 U.S.C. § 102(b) as being anticipated by Oles (U.S. Patent No. 4,145,451).**

Claims 64-70 were rejected under 35 U.S.C. § 102(b) as being anticipated by Oles. In particular, the Office Action asserts that the transitional language "consisting essentially of" in claim 64 does not exclude the additional components taught by Oles. Applicants respectfully traverse this rejection and request reversal by the Board.

Oles teaches a variety of chemical preservatives for foods. In each example, the base preservative includes acetic acid, phosphoric acid, and "moisture." However, the ingredient ranges in each example necessarily require additional components, i.e., no embodiment is identified as having acetic acid, phosphoric acid, and moisture components that add up to 100%. As a result, additional *food* ingredients, e.g., oil, sweetner, egg yolk, salt, starch, gum, spice, must be included.

### **- Claims 64-66**

The transitional phrase "consisting essentially of" limits the scope of a claim to the specified materials or steps "and those that do not materially affect the basic and novel characteristic(s)" of the claimed invention (*see* M.P.E.P. § 2111.03). The Board of Patent Appeals and Interferences' has stated that "[t]o determine the steps included versus excluded [where the transitional language 'consisting essentially of' is used,] the claim must be read in light of the specification," (MPEP § 2111.03 citing Ex Parte Hoffman, 12 USPQ2d 1061, 1063-64 (1989)).

The specification makes clear that Applicants intended to provide a cleaning composition for use in semiconductor integrated circuit (IC) fabrication consisting essentially of a dilute

aqueous solution of phosphoric acid and acetic acid, wherein, at least in one embodiment, the phosphoric acid is of a concentration of about 5% by volume or less and the acetic acid is of a concentration of about 30% by volume or less. It is also clear from the specification and the transitional language that other components that would materially alter the claimed composition's ability to function as a semiconductor cleaning agent are to be excluded. Such other components would most certainly include items considered contaminants in IC fabrication (e.g., oil, egg yolk, salt, starch, gum, spice, and the other ingredients that are inherently included in the Oles preservatives).

The Examiner asserts that Applicants have not provided any proof that the additional ingredients of Oles would be detrimental to the claimed invention (*see Office Action of January 31, 2003*, page 3). While it is understood that the burden rests with Applicant to establish that the additional food components of Oles would materially change the characteristics of the claimed invention, it is submitted that any person of ordinary skill in the art would clearly recognize that the inclusion of the food components taught by Oles in the claimed cleaning composition would materially affect the latter's ability to function as an IC cleaning composition.

For at least these reasons, Applicants respectfully submit that no legally sufficient basis is identified to reject claim 64 as anticipated by Oles. Claims 65-67 are also novel in view of Oles as they depend from claim 64 and further point to additional features of Applicants' invention. As a result, review and reversal of this rejection by the Board are respectfully requested.

- Claims 67-70

Claim 67 recites a cleaning composition for use in semiconductor IC fabrication including phosphoric acid and acetic acid, wherein the composition includes phosphoric acid at a concentration of about X% by volume or less, where X is 5, and acetic acid at a concentration of about (100-X)% by volume or less, wherein the composition is operable to remove exposed metal from a surface at a removal rate of less than about 200 Å/minute.

Claim 67 does not recite the transitional language "consisting essentially of." It does, however, recite a composition that includes phosphoric acid (at a concentration of about X% by volume or less, where X is 5) and acetic acid (at a concentration of about (100-X)% by volume or less). Moreover, it is recited within the claim that the composition is operable to remove exposed metal from a surface at a removal rate of less than about 200 Å/minute.

There is no teaching or suggestion identified in Oles of a composition having the claimed properties, e.g., no Oles composition is identified that is operable to remove material at the rate recited in claim 67. Moreover, the Examiner has provided no proof that a composition that includes the unclaimed Oles food components would inherently clean a surface, let alone at the claimed material removal rate.

"A claim is anticipated only if each and every element as set forth in the claim is found, either expressly or inherently described, in a single prior art reference." M.P.E.P. § 2131 (Eighth edition, Rev. 1, 2003) citing *Verdegaal Bros. v. Union Oil Co. of California*, 814 F.2d 628, 631, 2 USPQ2d 1051, 1053 (Fed. Cir. 1987). Moreover, "[t]he identical invention must be shown in as complete detail as is contained in the . . . claim." *Id.* at § 2131 citing *Richardson v. Suzuki Motor Co.*, 868 F.2d 1226, 1236, 9 USPQ2d 1913, 1920 (Fed. Cir. 1989).

As Oles fails to teach each and every element of claim 67, it fails to anticipate the claim. Consideration and reversal of the rejection of claim 67 (and the claims that depend therefrom) are, therefore, requested.

## **II. Whether claims 64-70 are properly rejected under 35 U.S.C. § 102(b) as being anticipated by Baker (U.S. Patent No. 5,508,229).**

Baker is directed to a method of forming solder bumps on a semiconductor wafer. To that end, Baker specifies an etching solution of "1 to 25% phosphoric acid by volume, 1 to 10% acetic acid by volume, 0.1 to 2% hydrogen peroxide by volume, and the remaining balance 63 to 98% deionized (DI) water by volume," (col. 4, lines 55-61). Thus, Baker teaches only solutions having some concentration of hydrogen peroxide.

- Claims 64-66

Independent claim 64 recites a cleaning composition "consisting essentially of a dilute aqueous solution of phosphoric acid and acetic acid, wherein the phosphoric acid is of a concentration of about 5% by volume or less and the acetic acid is of a concentration of about 30% by volume or less."

Baker explicitly includes hydrogen peroxide, a very strong oxidizing agent. Such a strong oxidizing agent is critical to Baker as the Baker etching solution must remove a 4 kÅ aluminum layer, a 2 kÅ nickel-vanadium layer, and a 4 kÅ copper layer during a single etching step.

The claimed cleaning composition has a chemistry that desirably prevents substantial removal of material from the surface being cleaned, i.e., the claimed composition is intended to clean organic and inorganic residue from a surface, not etch substantial amounts of material therefrom.

As a result, Applicants submit that the transitional language "consisting essentially of" in claim 64 excludes a component such as hydrogen peroxide as inclusion of such an ingredient would materially affect the basic characteristics of the claimed cleaning composition. Reconsideration and reversal of the rejection of claim 64 are therefore requested. Allowance of claims 65-66 are also requested not only due to their dependence on claim 64, but also because of the subject matter recited therein.

- Claims 67-70

Independent claim 67 recites "a cleaning composition for use in semiconductor integrated circuit fabrication comprising phosphoric acid and acetic acid, wherein the composition includes phosphoric acid at a concentration of about X% by volume or less, where X is 5, and acetic acid at a concentration of about (100-X)% by volume or less, and wherein the composition is operable to remove exposed metal from a surface at a removal rate of less than about 200 Å/minute.

Applicant submits that Baker fails to teach each and every element of claim 67 as required for anticipation. For example, Baker fails to teach a removal rate of less than about 200 Å/minute. Instead, Baker describes removal rates that vary from 1,000 Å/minute (when removing the 10,000 Å-thick layers of Al, NiV, and Cu in 10 minutes) to 6,700 Å/minute (when removing the same layers in 1.5 minutes). *See* col. 5, lines 27-37.

As Baker fails to teach at least this claim element, it cannot anticipate claim 67 and the claims that depend therefrom. Consideration and reversal of the rejection are requested.

**III. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Hitachi (JP 77048059) in view of Hwang et al. (U.S. Patent No. 4,681,657).**

Hitachi describes an etching process for etching aluminum layers. As a result, Hitachi utilizes an etchant including a high percentage of phosphoric acid, e.g., the described embodiment includes 75cc phosphoric acid, 15cc acetic acid, and 5cc water.

Hwang et al. is also directed to an etchant for resistivity-specific etching of doped silicon films. While admitting that Hitachi fails to teach the claimed concentrations, the Examiner has stated (*see Office Action of Sept. 16, 2002*) that Hwang et al. supports "the obviousness of diluting etching compositions" (*Id.* at paragraph 15) and cites the following portion of the Hwang et al. specification:

Initial attempts to produce a slower resistivity specific etch consisted of diluting the 1:3:8 HNA etch (Dash etch) with acetic acid while maintaining the molecular ratio of the oxidizing agent (HNO<sub>3</sub>) to the reducing agent (HF) at a constant value of 1.61 as in Dash etch. Whereas dilution did reduce the etch rate as expected, it also had undesirable side effects. The etchant was too preferential and failed to etch the higher doped polysilicon at the interface between the polysilicon layer and the underlying intrinsic or lightly doped surface. This resulted in a polysilicon residue remaining after etch. Not only was the etching non-uniform, the solutions were plagued by variable incubation periods before the onset of etching. This resulted in variation in the time required to remove polysilicon films of constant thickness. The result of these experiments is summarized in Table I.



*Hwang et al.*, column 5, lines 6-24.

To establish a *prima facie* case of obviousness, three basic criteria must be met. First, there must be some suggestion or motivation, either in the references themselves or in the knowledge generally available to one of ordinary skill in the art, to combine the references. Second, there must be a reasonable expectation of success. Finally, the prior art documents must teach or suggest all the claim limitations. See MPEP § 2143.

- Claims 64-66

Independent claim 64 recites a cleaning composition consisting essentially of a dilute aqueous solution. . . Thus, to render claim 64 obvious, Hitachi in combination with Hwang et al. must teach a dilute aqueous solution as defined in the specification. "Dilute" refers to a "composition having about 35% reactive components or less by volume" (*Specification*, page 6, lines 26-27).

Hitachi clearly fails to teach or suggest the composition of claim 64. In fact, during the prosecution of the present application's parent, (App. Ser. No. 09/325,375), the Patent Office concluded that Hitachi "clearly [failed] to meet the applicant's limitation of a 'dilute composition.'" See the '375 Office Action dated 16 January 2001 (paper no. 7, page 2, paragraph 2). Moreover, even though directed to an anticipation rejection, the Office Action in the '375 case concluded that Hitachi failed "to provide *motivation* for the modification of the solution to meet the applicant's limitation to a 'dilute composition'" (emphasis added).

However, the present Office Action asserts that, when combined with the identified teachings of Hwang et al., "a chemist's first attempt at reducing etching rate would be the obvious choice of dilution." Applicants submit that Hwang et al., however, offers no motivation to dilute the composition of Hitachi, especially to anywhere near the concentrations of the claimed invention. Thus, the combination of Hwang et al. with Hitachi is insufficient to render claims 64-66 *prima facie* obvious.

Hitachi requires a high concentration of phosphoric acid to ensure adequate metal removal, e.g., it must *not be* "dilute" as defined and claimed in the present application, to achieve its desired etching effect.

The identified portions of Hwang et al., moreover, do not motivate one to dilute the compositions of Hitachi to the claimed levels. In fact, the cited portions of Hwang et al. illustrate various negative effects of diluting the 1:3:8 HNA etch with acetic acid (e.g., too preferential, failed to etch effectively, residue resulting).

This distinction is important as the present invention is *not* directed, as alleged, to a diluted etching composition but rather is directed to a *composition useful for cleaning in IC fabrication*. While variation in concentration of chemical compositions may be common, there is no suggestion identified in either Hitachi or Hwang et al. to dilute the Hitachi composition to anywhere approaching the claimed concentrations, i.e., there is no suggestion that such a diluted composition would be an effective etchant. Further, there is no suggestion identified in Hitachi or Hwang et al. that reducing the phosphoric acid component of Hitachi to the ranges claimed by Applicants would yield a composition useful for *cleaning* a surface rather than etching material therefrom as intended. If anything, these documents suggest the opposite, i.e., much higher concentrations are advantageous as they are beneficial to their intended function of material removal.

Applicants further note that compositions having phosphoric acid in concentrations greater than that claimed (i.e., concentrations identified in Hitachi) may be detrimental to the function of the claimed cleaning composition. For example, the specification notes that "[if] the concentrations of the phosphoric acid component is increased above 5%, excess metal loss may undesirably result from the surface being cleaned" (see page 7, lines 17-18). Such material removal is thus contrary to the present invention but is essential to both Hitachi and Hwang et al.

Thus, the specification clearly indicates the detrimental effects of compositions which have high concentrations of phosphoric acid, e.g., those compositions described in Hitachi and Hwang et al. Yet no motivation is identified in Hwang et al. to dilute the phosphoric acid content of Hitachi to anywhere approaching the claimed concentrations.

For at least these reasons, the combination of Hitachi with Hwang et al. fails to support a *prima facie* obviousness rejection. Reconsideration and reversal of the rejection of claim 64 are therefore requested. Reconsideration and allowance of dependent claims 65-66 are also requested as these claims are submitted to be nonobvious both in view of their dependence on claim 64 and further in view of the subject matter addressed therein.

- Claims 67-70

In order to render claim 67 obvious, Hitachi, in combination with Hwang et al., must teach or suggest a cleaning composition of phosphoric acid at a concentration of about X% by volume or less, where X is 5, and acetic acid at a concentration of about (100-X)% by volume or less, wherein the composition is operable to remove exposed metal from a surface at a removal rate of less than about 200 Å/minute.

Hitachi clearly fails to teach or suggest the composition of claim 67, e.g., the phosphoric acid concentration of Hitachi is very high (about 79%) to ensure adequate etching while claim 67 recites a phosphoric acid concentration of about 5% by volume or less. Moreover, there is no suggestion identified in Hwang et al. to reduce the phosphoric concentration of Hitachi to a level such as that claimed as this would reduce Hitachi's etching effectiveness. In fact, the identified portion of Hwang et al. suggests that diluting etching component(s) adversely affected the composition in unintended ways. In addition, nothing is identified in either Hitachi or Hwang et al. that teaches or suggests the removal rate recited in claim 67.

For at least these reasons, claims 67-70 are not rendered obvious by the combination of Hitachi and Hwang et al. Reconsideration and reversal of the rejection are requested.

**IV. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Petzow (*Metallographic Etching*) in view of Hwang et al.**

- Claims 64-70

Claims 64-70 were rejected under 35 U.S.C. § 103(a) as being unpatentable over Petzow in view of Hwang et al.

Petzow teaches an etchant for etching materials where the etchant composition includes 10 ml of glacial acetic acid and 10 ml of phosphoric acid (i.e., 50%; 50%).

Applicants submit that the combination of Petzow in view of Hwang et al. fails to render the claims obvious for the same reasons discussed above with respect to the 35 U.S.C. § 103(a) rejection of claims 64-70 over Hitachi in view of Hwang et al. For example, Petzow requires a high concentration of phosphoric acid. Nothing in Hwang et al. would suggest reducing the phosphoric acid content of Petzow to the claimed levels of about 5% by volume or less. In fact, Hwang et al. indicates concentration reduction adversely affects etching characteristics. Moreover, with respect to claim 67, no teaching is identified in either Petzow or Hwang et al. of the recited removal rate.

For at least these reasons, Applicants submit that the combination of Petzow and Hwang et al. fails to render the claimed invention obvious. Reconsideration and reversal of the rejection are requested.

**V. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Koike et al. (U.S. Patent No. 4,256,520) in view of Hwang et al.**

- Claims 64-70

Claims 64-70 were rejected under 35 U.S.C. § 103 (a) as being unpatentable over Koike et al. (U.S. Patent No. 4,256,520) in view of Hwang et al.

Koike et al., teaches an etchant for removing gallium from a surface. The etchant includes high concentrations of phosphoric acid, acetic acid, and nitric acid. For example, Koike et al. states that possible ranges of components include:

phosphoric acid (98% concentration) . . . 6 to 4 parts in volume;

acetic acid (glacial acetic acid) . . . 5 to 2 parts in volume; and  
nitric acid (specific gravity: 1.38) . . . 3 to 0.1 parts in volume.  
*Col. 2, line 61 - col. 3, line 5.*

As the previous Office Actions admit, Koike et al. does not teach the dilute aqueous solution having the claimed concentrations recited in claim 64. Rather, Koike et al. teaches only solutions with a high phosphoric acid content. Thus, for the same reasons discussed above with respect to the rejection over Hitachi in view of Hwang et al., *prima facie* obviousness with respect to claims 64-70 is unsupported. Further, nothing is identified in Koike et al. or Hwang et al. that suggests either the phosphoric acid concentration or the claimed removal rate recited in claim 67. For at least these reasons, reconsideration and withdrawal of the rejection are requested.

**VI. Whether claims 64-70 are properly rejected under 35 U.S.C. § 103(a) as being unpatentable over Martin et al. (U.S. Patent No. 4,230,522) in view of Hwang et al.**

- *Claims 64-70*

Claims 64-70 were rejected under 35 U.S.C. § 103 (a) as being unpatentable over Martin et al. (assumed to be U.S. Patent No. 4,230,522) in view of Hwang et al.

Martin et al., teaches an etchant for removing thin films of aluminum, silicon, and aluminum-silicon-alloy on composite structures. The identified embodiment of the etchant includes a high concentration of phosphoric acid and also includes ammonium fluoride and nitric acid (see, e.g., Example 9, wherein the etchant used a mixture of concentrated phosphoric acid (85%), glacial acetic acid, water, concentrated ammonium fluoride (40%) and concentrated nitric acid (70%) in the proportions of 76:15:5:2:3, respectively). Thus, as the Office Action admits, Martin et al. does not teach the claimed concentrations. As a result, for the same reasons discussed above with respect to the rejection over Hitachi in view of Hwang et al., *prima facie* obviousness is unsupported with respect to claims 64-70. For at least these reasons, reconsideration and reversal of the rejection are requested.

## Conclusion

For the reasons provided herein, Applicants respectfully submit that pending claims 64-70 are allowable in view of the cited art. Review and reversal of the rejections are respectfully requested.

Respectfully submitted,

Hineman et al.,

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30 June 2003

Date

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The undersigned hereby certifies that this paper is being deposited with the United States Postal Service "Express Mail Post Office to Addressee" service under 37 CFR §1.10 on the date indicated above and is addressed to Mail Stop Appeal Brief-Patents, Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

By: Sara E. Olson

Name: SARA E. OLSON

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## Appendix A - Appeal Brief

Ser. No. 09/935,234

Attorney Docket No. 150.00450122

### DILUTE CLEANING COMPOSITION AND METHOD FOR USING SAME

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64. A cleaning composition for use in semiconductor integrated circuit fabrication, the cleaning composition consisting essentially of a dilute aqueous solution of phosphoric acid and acetic acid, wherein the phosphoric acid is of a concentration of about 5% by volume or less and the acetic acid is of a concentration of about 30% by volume or less.

65. The cleaning composition of claim 64, wherein the phosphoric acid is of a concentration of about 5% or less by volume and the acetic acid of a concentration of about 10% or less by volume.

66. The cleaning composition of claim 64, wherein the phosphoric acid is of a concentration of about 5% or less by volume and the acetic acid is of a concentration in the range of about 20% by volume to about 30% by volume.

67. A cleaning composition for use in semiconductor integrated circuit fabrication comprising phosphoric acid and acetic acid, wherein the composition includes phosphoric acid at a concentration of about X% by volume or less, where X is 5, and acetic acid at a concentration of about (100-X)% by volume or less, and wherein the composition is operable to remove exposed metal from a surface at a removal rate of less than about 200 Å/minute.

68. The cleaning composition of claim 67, wherein the composition is a dilute composition, wherein the dilute composition includes phosphoric acid at a concentration of about 5% by volume or less, acetic acid at a concentration of about 30% by volume or less, and deionized water.

69. The cleaning composition of claim 68, wherein the dilute composition includes phosphoric acid at a concentration of about 5% by volume or less, acetic acid at a concentration of about 10% by volume or less, and deionized water.

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70. The cleaning composition of claim 68, wherein the dilute composition includes phosphoric acid at a concentration of about 5% by volume or less, acetic acid at a concentration in the range of about 20% by volume to about 30% by volume, and deionized water.